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खाद्य पदार्थ में डाईमेथोएट के अंश ज्ञात करने की विधि

(पहला पुनरीक्षण)

Indian Standard

METHODS FOR DETERMINATION OF DIMETHOATE RESIDUES IN FOOD COMMODITIES

(First Revision)

ICS 65.100.10

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

June 2004

Price Group 2

Pesticides and Pesticide Residue Analysis Sectional Committee, FAD 1

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides and Pesticide Residue Analysis Sectional Committee had been approved by the Agricultural and Food Products Division Council.

This standard was first published in 1984.

Dimethoate formulations are extensively used in agriculture for the control of many insect pests. Assessment of pesticides residues in food commodities is therefore an important step in safeguarding human health.

In the preparation of this standard, due consideration has been given to the limits of dimethoate residues laid under the provisions of *Prevention of Food Adulteration Act*, 1955 and the test methods are sensitive to the prescribed level of residues.

This standard will enable the health authorities and others engaged in the field to follow uniform test procedure for the estimation of dimethoate residues in food commodities.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS FOR DETERMINATION OF DIMETHOATE RESIDUES IN FOOD COMMODITIES

(First Revision)

1 SCOPE

1.1 This standard prescribes spectrophotometric and gas chromatographic methods for the determination of dimethoate [O,O-dimethyl-S-(N-methyl carbamoyl methyl) phosphorodithioate] residues in foods.

1.1.1 The spectrophotometric method may be adopted as a limit test for routine purposes and is generally applicable at 0.1 μg/g (0.1ppm) residue level, whereas, gas chromatographic method shall be the reference method with the limit of detection of 0.02 to 0.01 μg/g (0.02-0.01 ppm).

1.1.2 Though no set procedure for thin layer chromatography (TLC) is being prescribed, standardized TLC procedures may be followed, if necessary for the purpose of clean up, identification and confirmation of dimethoate residues.

2 REFERENCES

The following Indian Standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
11380 (Part 1) : 1985	Methods of sampling for determination of pesticides residues: Part 1 Agricultural and food commodities

3 SAMPLING

The representative samples for the purpose of estimating dimethoate residues in foods shall be drawn in accordance with IS 11380 (Part 1).

4 PREPARATION OF SAMPLES

4.1 Apparatus

4.1.1 *Waring Blender or Equivalent*

4.1.2 *Rotary Vacuum Evaporator*

4.1.3 *Chromatographic Column* — 30 cm long, 1.5 cm inner diameter.

4.1.4 *Buchner Funnel*

4.1.5 *Air Blower*

4.1.6 *Water Bath*

4.1.7 *Laboratory Shaker*

4.2 Reagents

4.2.1 *Methylene Chloride* — Analytical reagent grade, glass redistilled.

4.2.2 *Anhydrous 3A Alcohol* — Mix 95 ml of redistilled absolute ethanol with 5ml of redistilled absolute methanol.

4.2.3 *Propylene Glycol*

4.2.4 *Chromatographic Adsorbent* — Silica gel, column chromatography grade (60-120 mesh).

4.2.5 Activated Carbon

4.2.6 *Sodium Sulphate* — Anhydrous.

4.2.7 *Hyflo Super-cel.*

4.2.8 *Methanol* — Analytical reagent grade, glass redistilled.

4.2.9 *n-Hexane* — Analytical reagent grade, glass redistilled.

4.2.10 *Glycerine* — 0.6 percent (*v/v*) in 3A alcohol.

4.3 Extraction

4.3.1 *Fruits and Vegetables*

4.3.1.1 Transfer 50 g of the finely chopped fruit or vegetable sample into the blender jar. Add 200 ml of redistilled methylene chloride and 60 g of anhydrous sodium sulphate and blend at high speed for 5 min. Filter the extract through a Buchner funnel attached to 500 ml filter flask containing Whatman No.1 or equivalent filter paper overlaid with 0.5 cm layer of hyflo super-cel or equivalent filter aid. While filtering, apply vacuum cautiously until all the solution has percolated into the filtering flask. Return the filter cake back to the blender jar and blend further with 100 ml of redistilled methylene chloride and filter as before. Wash the blender jar and the filter cake twice with 50 ml portions of redistilled methylene chloride. The filtrate and washings collected in the filtering flask are further subjected to clean up step in accordance with **4.4**.

4.3.2 *Grains, Dry Fruits, Tea and Cured Tobacco*

4.3.2.1 Transfer 50 g of the finely powdered sample into a 500 ml stoppered conical flask, add 200 ml of redistilled methylene chloride and shake vigorously for 1h, allow to stand overnight and transfer the mixture to the waring blender jar and carry out extraction as in **4.3.1.1**.

4.3.3 *Oilseeds*

Transfer 50 g of the sample to waring blender jar, add 200 ml methylene chloride and blend at high speed for 5 min. Transfer the macerate to a 500 ml conical flask and allow to stand overnight. Transfer the mixture to the waring blender jar and carry out extraction as in **4.3.1.1**.

4.4 Clean-Up

4.4.1 To the filtrate obtained after extraction, as in **4.3**, add 1.0 g of activated charcoal, shake vigorously for 2 min, allow to stand for 2 min and filter through Whatman No.1 or equivalent filter paper overlaid by a layer of anhydrous sodium sulphate and mounted on a Buchner funnel, collecting the filtrate in a 500 ml filtering flask. Transfer the filtrate to a rotary vacuum evaporator, add 3 drops of propylene glycol and concentrate the extract to about 1 to 2 ml. During evaporation the temperature of the water bath should not be more than 40°C. To the residue in the evaporator flask add 50 ml of 15 percent aqueous methanol shake vigorously and transfer the solution to a 125 ml separating funnel. Wash the flask twice with 5 ml portions of 15 percent aqueous methanol transferring the washings to the separating funnel. Add 40 ml of *n*-hexane to the separating funnel, shake well for 2 min and allow the phase to separate. Discard the upper hexane phase and wash the aqueous phase twice more with 10 ml portions of *n*-hexane discarding the hexane washings. Extract the aqueous layer thrice with 20 ml portions of methylene chloride, combine the methylene chloride extracts and concentrate to about 3 to 5 ml in a rotary vacuum evaporator.

4.4.2 Prepare a chromatographic column containing 10 g of silica gel using methylene chloride as the solvent. Transfer the methylene chloride solution obtained in **4.4.1** quantitatively on to the column using 3-5 ml of methylene chloride. Elute the column with methylene chloride. Collect about 200 ml eluate in 250 ml round bottom flask, add 10 ml of 0.6 percent (*v/v*) glycerine solution in 3A alcohol and evaporate the solvent in a rotary vacuum evaporator.

5 SPECTROPHOTOMETRIC METHOD

5.1 Principle

Dimethoate is subjected to alkaline hydrolysis and the resultant methylamine is determined spectrophotometrically after reaction with 1-chloro-2, 4-dinitrobenzene, and measuring the absorbance at 520 nm.

5.2 Apparatus

5.2.1 *Spectrophotometer*

5.2.2 *Water Bath,* maintained at 60 ± 20°C

5.3 Reagents

5.3.1 *Methanolic Sodium Hydroxide* — Dissolve 2 g of sodium hydroxide in 100 ml of absolute methanol.

5.3.2 *Chlorodinitrobenzene Solution* — Recrystallize 1-chloro-2, 4-dinitrobenzene by dissolving it in a minimum volume of 3A alcohol, filtering the solution and addition of distilled water to get crystals. Again filter, wash with distilled water and dry in a desiccator. Prepare a 2 percent solution by dissolving 0.2 g of this purified material in 10 ml of anhydrous 3A alcohol. Prepare fresh solution each time.

5.3.3 *Standard Dimethoate Solution* — Dissolve 10 mg of dimethoate (99 percent pure) and make up to 100 ml in redistilled methylene chloride. Transfer 10 ml of this solution to a second 100 ml volumetric flask and make up to the mark with methylene chloride. This standard solution contains 10 µg dimethoate per millilitre.

5.4 Estimation of Dimethoate Residues

5.4.1 Dissolve and quantitatively transfer the residue obtained in **4.4.2** using 15 ml of 3A alcohol, to a 25 ml volumetric flask, add 2 ml of methanolic sodium hydroxide solution and proceed for hydrolysis and colour development as given under **5.4.2**.

5.4.2 *Preparation of Standard Curve*

Pipette 0, 0.5, 1.0, 2.5, 5.0, 7.5 and 10.0 ml of the standard (10 µg/ml) solution into a series of 150 ml of round bottom flasks. Dilute to 60 ml with methylene chloride, add 10 ml of 0.6 percent glycerine solution in 3A alcohol and evaporate the solvent in a rotary vacuum evaporator keeping the flask in a water bath maintained at 40°C.

Transfer the residue with 15 ml of 3A alcohol quantitatively to a 25 ml volumetric flask, add 2 ml of methanolic sodium hydroxide solution, stopper the flask and hydrolyze by keeping the flasks in a water bath at 60°C for 10 min with occasional shaking. Cool the flasks and add 0.5 ml of chlorodinitrobenzene reagent. Mix the contents and make up to the volume with 3A alcohol. Read the absorbance of the resultant solution at 520 nm; against 3A alcohol as reference. Prepare the standard curve by plotting the absorbance against µg of dimethoate.

5.5 Expression of Results

Report dimethoate residues in the sample by using the standard curve in terms of µg/g (ppm).

6 GAS CHROMATOGRAPHIC METHOD

6.1 Principle

The dimethoate residue extracted from samples is dissolved in acetone and estimated by gas chromatograph equipped with thermionic or flame photometric detector. The content of dimethoate is determined by comparing the response with the response for a known standard of similar concentration.

6.2 Apparatus

6.2.1 *Gas Chromatograph* — A gas chromatograph equipped with thermionic or flame photometric detector is operated under the following suggested parameters. These parameters may be varied as per available facilities provided standardization is done.

Column : Glass, 60 cm long and 4 mm i.d., packed with either 5 percent OV – 101 or DC 200 – on 60-80 mesh gas chrom Q
Column temperature : 165°C
Injection port temperature : 225°C
Detector temperature : 200°C
Gas flow rates : Nitrogen, 40 ml/min
Hydrogen, 15 to 18 ml/min
Air, 150 ml/min
Recorder chart speed : 1 cm/min
Retention time : 2.5 min approximately

6.3 Reagents

6.3.1 *Acetone* — Analytical reagent grade, glass redistilled.

6.3.2 *Standard Dimethoate Solution* — Dissolve 10 mg dimethoate (pure) and make up to 100 ml in redistilled acetone. Prepare working standard solution of required concentration by diluting suitably an aliquot of the above solution with redistilled acetone.

6.4 Procedure

Dissolve the concentrated residue (*see* **4.4.2**) in 5 ml acetone and inject 2.0 µl of this solution into the gas chromatograph. Identify the dimethoate peak by its retention time and measure the peak area.

6.5 Calculation

$$\text{Dimethoate residue, µg/g (ppm)} = \frac{A_s}{A_{std}} \times \frac{M}{M_1} \times \frac{V}{V_1} \times f$$

where

A_s = peak area of sample;
M = mass, in µg, of standard injected;
V = final volume of sample in ml;
A_{std} = peak area of the standard;
M_1 = mass, in g, of the sample;
V_1 = µl of sample injected; and
F = recovery factor $= \dfrac{100}{\text{Percent mean recovery}}$

NOTE — Percent mean recovery is determined by taking untreated control sample to which known amount of dimethoate is added and analyzed as described above.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

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